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Abstract:	Poly-Ether-Ether-Ketone (PEEK) has been widely used for tribological applications with steel counterparts because of its superior mechanical and self-lubricating properties. Lubrication has the potential to further improve its performance. However, lubrication can have positive or negative effects depending on the operating conditions and little is known about the mechanism of lubrication. To elucidate these aspects, the friction and wear properties of the PEEK/steel contact in both dry and poly-α-olefin (PAO) lubricated conditions were investigated by tribological tests and surface analytical techniques. The nanoindentation measurements showed that lubrication with PAO had a softening effect on the wear track of PEEK, but no correlation was established with the wear of PEEK. The tribological behavior, for both dry and lubricated contacts, was correlated with the formation of PEEK transfer films on the steel counterparts. The thickness of these films, as determined by Electron Probe Micro Analysis (EPMA) and X-ray Photoelectron Spectroscopy (XPS), was controlled by the rates of their transfer and removal and depended on the operating conditions. Moreover, lubrication with PAO not only inhibited the transfer but also suppressed the removal of PEEK films. This study thus sheds light on the mechanism of lubrication of the PEEK/steel contact.
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Effect of lubrication on friction and wear properties of PEEK

with steel counterparts

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Abstract

Poly-Ether-Ether-Ketone (PEEK) has been widely used for tribological applications with steel counterparts because of its superior mechanical and self-lubricating properties. Lubrication has the potential to further improve its performance. However, lubrication can have positive or negative effects depending on the operating conditions and little is known about the mechanism of lubrication. To elucidate these aspects, the friction and wear properties of the PEEK/steel contact in both dry and poly-α-olefin (PAO) lubricated conditions were investigated by tribological tests and surface analytical techniques. The nanoindentation measurements showed that lubrication with PAO had a softening effect on the wear track of PEEK, but no correlation was established with the wear of PEEK. The tribological behavior, for both dry and lubricated contacts, was correlated with the formation of PEEK transfer films on the steel counterparts. The thickness of these films, as determined by Electron Probe Micro Analysis (EPMA) and X-ray Photoelectron Spectroscopy (XPS), was controlled by the rates of their transfer and removal and depended on the operating conditions. Moreover, lubrication with PAO not only inhibited the transfer but also suppressed the removal of PEEK films. This study thus sheds light on the mechanism of lubrication of the PEEK/steel contact.

1. Introduction

Polymers and polymer-based composites are becoming preferred materials in many tribological applications. Compared with metals, polymer materials have advantages such as lightweight, reduced noise and self-lubricating properties which make their use in automotive, aerospace, medical, industrial applications highly desirable [1–9]. On the other hand, the mechanical strength and thermal stability of polymers are lower than those of metals, and therefore they tend to suffer from failures such as wear, local melting and pitting when used in severe conditions [10–13].

Among the many types of polymers, Poly-Ether-Ether-Ketone (PEEK) has suitable properties for tribological applications under severe conditions due to its superior mechanical properties and higher thermal stability than those of other conventional polymers [1,3,14]. PEEK is commonly used with steel counterparts, and numerous studies have investigated the friction and wear properties of PEEK/steel contacts in dry conditions [15–21]. One of the key factors of the favorable tribological properties in dry conditions is the formation of transfer films of PEEK on steel counterparts [19,22]. They act as protective films avoiding the direct contact of PEEK with hard asperities of steel and thus reduce friction and wear. In-situ observation of the PEEK wear process was reported by Puhan and Wong [23] and suggested that PEEK wear debris ploughed by the asperities of the counter surface re-entered the contact and became trapped between asperities of the counter surfaces by compression, forming PEEK transfer films on the counter surfaces. Kalin et al. [24] reported transfer-film thickness and coverage showed good correlation with the friction and wear performances of PEEK and PEEK composites.

While there are many attempts to improve the tribological performance of PEEK from the material side, as in the case of PEEK composites with specific fillers [24–29], lubrication also has the potential to further reduce friction and wear of PEEK. Zhang et al. [30] reported lubrication with diesel fuel decreased friction and wear of PEEK in a PEEK plate/steel ring sliding contact while observing the transfer film on steel counterparts. However, compared to dry conditions, little work has been reported on the tribological performance of PEEK in lubricated contacts and most of them were focused on water lubrication [31,32]. Additionally, it was shown by Kurdi et al.[33] that lubrication can have a negative effect on the wear of PEEK. In the contact between a PEEK pin and a steel disc, lubrication with water reduced friction, but increased the wear of PEEK dramatically compared with dry conditions. As a PEEK transfer film on the steel counterpart was not observed in water lubricated condition the authors concluded that water lubrication inhibits the formation of stable transfer films on steel counterparts. Yamamoto and Takashima [34] reported a similar acceleration of wear of PEEK in water lubrication. Additionally, the hardness of the PEEK sliding surface decreased in water lubrication, while the mere immersion in water did not lead to any changes. The authors postulated that the softening of the rubbing surface during

water lubrication may be due to the interaction between water molecules and the carbonyl group in the PEEK molecular structure and was considered to be the key factor for the high wear of PEEK. Similar softening of PEEK in water lubricated conditions were reported by Yamaguchi and Hokkirigawa [35]. Some work on oil lubricated PEEK was also reported [36–38], but the key factors mentioned above were not fully investigated and the reason why lubrication can have positive or negative effects on wear of PEEK depending on tribological conditions remains unclear.

The current study aimed to investigate the effect of lubrication on tribological properties of PEEK and elucidate its mechanism of lubrication. The friction and wear behavior of PEEK was studied under dry and oil lubricated conditions in sliding-rolling contact with steel counterparts. Most of the previously reported work was carried out in pure sliding contacts. However, lubricated PEEK/steel contacts are envisaged in tribological applications such as gears and bearings that experience sliding-rolling contacts. The two most important factors influencing tribological properties, the PEEK transfer films on steel counterparts and modifications of PEEK hardness after tribological testing have been carefully investigated.

A better understanding of the effect and working mechanism of lubrication is essential to not only formulating optimal lubricants for PEEK applications but also developing more suitable systems PEEK applied.

2. Experimental Methods

2.1. Materials

PEEK plate specimens $40 \times 40 \times 2$ mm were injection molded from commercially available PEEK (Solvay® KT-820 NT). The Ra surface roughness of the PEEK plates was less than 0.05 μm and they were used without polishing. To evaluate the effect of lubrication on the tribological properties of PEEK in the boundary lubrication regime, half-inch steel balls (AISI 52100) were purchased from PCS instruments Ltd and shot blasted to Ra of approximately 0.5 μm. New pates and balls were used for each test and cleaned with a hydrocarbon-mix solvent (FASTCLEAN 201, CRC Industries UK Ltd) and isopropanol prior to the test. A poly-α-olefin (PAO) oil with a density of 0.819 g/cm^3 and viscosity of 17.5 cSt at 40 °C and 3.9 cSt at 100 °C (~31 cSt at 25 °C) was used as lubricant.

2.2. Tribological tests

Tribological tests were carried out using a Mini Traction Machine (MTM) from PCS instruments Ltd in dry and PAO lubricated conditions. The PEEK plate was placed on top of a bespoke MTM disc and secured in place with the nut (Fig. 1). The test conditions are summarized in Table 1. The steel ball and disc were driven independently to create

a mixed sliding-rolling contact with slide-roll ratios (SRRs) of 50 % and 100 %. All tests were carried out under an entrainment speed of 0.5 m/s and an applied load of 50 N for 60 minutes at ambient temperature (approximately 25 °C). The maximum Hertzian contact pressure (P_{max}) was calculated as 0.16 GPa using an elastic modulus of 3.83 GPa and Poisson's ratio of 0.33 for PEEK (adapted from supplier's catalogue). The wear tracks on the PEEK plates were analyzed at the end of the tribological tests to quantify wear and changes in hardness. The wear rates of PEEK plates were calculated from wear profiles measured with a stylus profilometer (Talysurf, Taylor Hobson).

2.3. Nanoindentation measurements

Nanoindentation measurements were performed on an iMicro nanoindenter (NANOMECHANICS, Inc.) equipped with a Berkovich tip. To evaluate the hardness of PEEK as a function of indentation depth, the continuous stiffness measurement (CSM) technique [39–41] was employed by applying a small, sinusoidally varying signal on top of a DC signal driving the indenter. The hardness at each indentation depth is determined by analyzing the response of amplitude and phase. The frequency and displacement amplitude values were 110 Hz and 1 nm, respectively. Under a load range of 50 mN, a target depth of 3 μm and a strain rate of 0.01 s^{-1}, sixteen points at 50 μm intervals were measured inside and outside of wear tracks for each PEEK plate. The average hardness at each indentation depth and the standard deviation were then calculated.

2.4. Surface Analyses (EPMA, XPS)

Electron Probe Micro Analysis (EPMA) and X-ray Photoelectron Spectroscopy (XPS) were carried out on the transfer films found on steel balls after tribological testing using a JXA-8530F (JEOL, Ltd.) and a PHI 5000 VersaProbe III (Ulvac-Phi, Inc.). Test specimens were rinsed with hydrocarbon solvent and dried before measurement. The EPMA carbon maps were acquired with a 15-kV beam at 100 nA current. The XPS spectra were obtained using a monochromatic A1 Kα source. The C 1s detailed scan was performed with a spot size of 100-μm diameter. In general, EPMA shows average surface elemental composition up to a few μm depth while XPS reflects the nature on the uppermost surfaces up to a few nm depth [42]. This makes both techniques suitable for investigation of thickness and chemical nature of transfer films.

3. Results and Discussion

3.1. Tribological tests

The different mechanical properties of the two materials, steel and PEEK which embodied the tribological contact along with the testing conditions employed in this study meant that the region of operation was borderline between piezoviscous-elastic and isoviscous-elastic. Therefore, the film thickness calculations give almost the same results

irrespective of using the equations for piezoviscous-elastic [43] or isoviscous-elastic lubrication [44,45].   The lubricated contacts at both SRRs (50 % and 100 %) experienced similar entrainment speeds (0.5 m/s) and therefore lubricant film thicknesses and Lambda ratios. The calculated value of approximately 0.2 for Lambda ratio entails that the contact operated in the boundary lubrication regime.

Friction coefficient values as a function of time and the averaged friction during the last ten minutes of testing for the dry and lubricated tests are illustrated in Fig. 2 and Fig. 3. Regardless of SRR, a significant friction reduction was achieved in the PAO lubricated tests compared to the dry ones. Unexpectedly, for both dry and lubricated contacts, friction was lower for 100 % SRR than for 50 % SRR.

The wear rates of PEEK plates in Fig. 3 show a different trend than friction coefficients. In dry conditions, the 100 % SRR wear rate was higher than at 50 % despite showing the lower friction. In the case of lubricated contacts, the wear trend was opposite the unlubricated ones, with 100 % SRR resulting in lower wear than 50 % SRR. Additionally, lubrication showed a negative effect on wear at 50 % SRR and a positive one at 100 % SRR and almost no correlation was seen between friction and wear results.

To investigate the wear mechanism of PEEK plates, optical images of the wear tracks under two magnifications were analyzed (Fig. 4(a-h)). The high magnification images show that the appearance of the wear tracks is different, especially in the case of two specimens: the dry 50 % SRR and PAO 50 % SRR. The wear track on the dry 50 % SRR plate showed signs of adhesive or fatigue wear. By contrast, the wear track of the PAO 50 % SRR showed the characteristic pattern of abrasion as observed by Schallamach for abrasive wear of rubber [46]. When tests were conducted with smooth steel balls and thus in the hydrodynamic lubrication regime, there was almost no wear on the PEEK plates regardless of SRR. Further aspects of the wear mechanisms will continue to be investigated in the following sections.

Previous research pointed out that lubrication can have an important effect on the hardness of PEEK and the formation of transfer films on steel counterparts. The current study decided to study the mechanism of lubrication of PEEK/steel contacts and thus understand their friction and wear behavior by focusing on these two key factors.

3.2. Nanoindentation investigations of PEEK hardness

Changes in PEEK hardness after testing were measured using nanoindentation. Hardness measurements were carried out on PEEK plates outside and inside of wear tracks and the hardness values were plotted as a function of indentation depth (Fig. 4(i-l)). In all tests, hardness showed high values at indentation depth values <0.5 µm. These values are thought to be attributed to a phenomenon known as indentation size effect (ISE), and not regarded as physically significant because they are distorted by the inadequacies in the procedures applied to provide corrections for the imperfections in the tip

geometry [41,47,48].

The hardness at 2 μm indentation depth for the four specimens are graphically summarized in Fig. 5. The hardness values for the specimens used in the dry 50 % and 100 % SRR tests are very similar inside and outside of wear track. By contrast, the PAO lubricated specimens show a significant decrease in hardness (of approximately 23 %) inside the wear tracks for both 50 % and 100 % SRR tests. This implies that lubrication with PAO reduced the hardness of the PEEK plates, regardless of SRR. Interestingly, lubrication with PAO (a non-polar solvent) caused softening of PEEK as in the case of lubrication with water (a polar solvent). The hardness of outside of the wear tracks showed almost the same value for all specimens. This indicates softening of PEEK did not occur just by immersing in PAO. Noticeably, the hardness of 50 % and 100 % SRR tests showed almost the same values in dry and lubricated conditions respectively, the opposite to wear rates. This implies softening of PEEK by lubrication with PAO was not the main cause of the high wear rate of the PEEK plate in the PAO SRR 50 % test. In fact, the PAO 100 % SRR test showed the lowest wear rate in spite of the decrease in the hardness of PEEK.

3.3. Investigations of PEEK transfer films on steel balls

The wear tracks of the steel balls were analyzed after tribological tests for wear and PEEK transfer films. Optical images of all specimens in Fig. 6(a-d) showed that there was almost no wear on steel balls. As the hardness of the steel ball is much higher than that of PEEK plates, it was expected that wear mainly occurs on the PEEK plates. The balls used in the dry tests show the presence of PEEK wear debris outside the wear tracks, especially abundant with 100 % SRR. In lubricated tests, the wear debris would be flushed out by PAO, but small amounts of wear debris were still observed as white residuals/marks just outside of wear tracks.

The visual evaluation of the amount of PEEK transfer films on the wear track was challenging so EPMA carbon mapping analysis was also employed. EPMA is equipped with Wavelength Dispersive X-ray spectroscopy (WDX) which counts the number of X-rays of a specific wavelength diffracted by a crystal, therefore it has higher resolution than Energy dispersive X-ray spectrometry (EDX), and is suitable for carbon mapping. EPMA carbon maps on after-test steel balls in Fig. 6(e-h) showed a clear difference between the steel ball wear tracks. The carbon amount is indicated by the colour scale on the map. A good correlation can be seen between the wear rates of PEEK plates (Fig. 3) and the carbon amount on the wear track of steel balls (Fig. 6(e-h)). A large amount of carbon as a uniform layer was detected on the wear tracks of the steel balls used in the dry 50 % SRR test and PAO 100 % SRR test. These testing conditions also led to low wear rates of PEEK plates. A reduced amount of carbon was detected on the steel ball wear track from the PAO 50 % SRR test which showed a very high wear rate of the PEEK plate. The wear track of the steel ball used in the dry 100 % SRR test

showed non-uniform coverage with carbon varying between high and low concentrations.

The presence of carbon detected with EPMA is supposedly related to PEEK transfer films on steel ball wear tracks but this assumption is questionable in the case of PAO lubricated balls. To confirm the assumption, XPS analysis of C 1s spectra was recorded on steel ball wear tracks. As Fig. 7 shows, C 1s spectra in all tests, except for the PAO 50 % SRR, showed almost identical peak curves which matched well the spectra recorded on an unused PEEK plate and the literature reported PEEK spectra [49]. A small difference in spectra is seen for the PAO 50 % SRR wear track which can be due to the very low carbon presence on the steel ball. Therefore, from the XPS results it can be concluded that EPMA carbon maps are a valid analysis of PEEK transfer films on steel ball wear tracks. Moreover, a good correlation is seen between the EPMA detected carbon amount on steel ball wear tracks and the wear rates of PEEK plates. This indicates that the presence of a PEEK transfer film on the steel ball is a key factor not just for dry conditions but also for lubricated ones.

The presence and thickness of PEEK transfer films on the steel counterpart is determined by the dynamic balance between film transfer and removal process and thus can be significantly influenced by the operating conditions (SRR). As the results showed, in dry conditions, the increase in SRR from 50 % to 100 % mean the higher shear stresses (higher sliding speed) accelerated film removal and led to a non-uniform transfer film as seen in the dry 100 % SRR test (Fig. 6(f)). Similar to the previous reports for water lubrication, PAO inhibited the formation of transfer films in the 50 % SRR test, causing direct contact between the PEEK surface and asperities of the counter steel surface. This explains the difference in surface patterns in Fig. 4. The dry 50 % SRR test shows a specific pattern on the PEEK plate (Fig. 4(e)) and a thick PEEK transfer film on the steel surface which bears the hallmarks of adhesive wear or fatigue wear that can take place in the PEEK/PEEK contact. By contrast, the PAO 50 % SRR test showed an abrasion pattern on PEEK (Fig. 4(g)) and a thin PEEK transfer film on the ball wear track indicating abrasive wear in the PEEK/steel contact which resulted in the most significant wear loss from all tests.

However, it was still unclear why the variation of SRR in the PAO lubricated tests resulted in significant differences in PEEK transfer film thicknesses. A plausible explanation could be that the SRR influences/controls the balance of film transfer and removal process and lubrication with PAO can not only inhibit the transfer but also suppress the removal of PEEK films. Therefore, additional tests were specifically devised to investigate this.

3.4. Additional tests using preconditioned steel balls

To investigate why poorer PEEK transfer films resulted from the dry 100 % SRR and PAO 50 % SRR tests additional testing was carried out using preconditioned steel balls. Run-in dry 50 % SRR tests were carried out for 10 minutes (testing conditions similar to those used for the previous dry tests) to achieve a uniform PEEK transfer films on the steel

ball wear track as seen in Fig. 6(e). The EPMA carbon map of the 10 minutes preconditioned steel ball is shown in Fig. 10(a). The preconditioned balls were then paired with unused PEEK plates and tested in dry 100 % SRR and PAO 50 % SRR for 60 minutes. The friction and wear results of these tests are shown in Fig. 8 and Fig. 9, in comparison of tests with new balls. The preconditioned balls did not affect friction and wear in the dry 100 % SRR test. The friction coefficients with the preconditioned ball started from lower values than those with the new ball but in a short time (less than 10 minutes) converged to the same level. The EPMA carbon map of the transfer film in Fig. 10(b) showed a non-uniform coverage of the wear track similar to the test with the new ball in Fig. 6(e). These results indicate that the transfer and removal of PEEK films in dry 100 % SRR tests were very fast and the preconditioned ball did not have a meaningful effect. By contrast, the preconditioning of the ball significantly reduced friction and wear in PAO 50 % SRR test as shown in Fig. 8 and Fig. 9. Friction coefficient values with the preconditioned ball were half those with the new ball for 20 minutes and stabilized at a lower level for the rest of the test. Moreover, the wear rate of the PEEK plate decreased drastically. The transfer film with preconditioning, Fig. 10(c), was slightly thicker than that with the new ball, Fig. 6(g). These results indicate that lubrication with PAO suppressed the speed of the removal of PEEK films, and thus the initial film formed through preconditioning the balls was reasonably stable during the test.

Results from the additional tests confirmed the following assumptions: (i) SRR affects the balance of transfer and removal of PEEK films and (ii) lubrication with PAO not only inhibits the transfer but also suppresses the removal of PEEK films from the steel counterparts.

3.5. Mechanism of lubrication

Similar to the previously reported research on water lubrication, the softening of PEEK was observed in this study, but no correlation was seen with wear behavior. On the other hand, the formation of a transfer film was found to be the dominant factor in this study and a schematic of the assumed mechanism of lubrication is presented in Fig. 11. Lubrication with PAO not only inhibits the transfer but also suppresses the removal of PEEK films, and the dynamic balance between these processes is controlled by SRR. A further similarity to water lubrication is that PAO inhibits the formation of a PEEK film on the steel counter surface, thus causing direct contact between PEEK and steel. The excessive wear rate in the PAO 50 % SRR test was the result of abrasive wear, inducing an abrasion pattern on the PEEK plate (Fig. 4(g)). On the other hand, the dynamic balance between transfer and removal significantly changes in the PAO 100 % SRR test. With a high SRR, the speeds of the transfer and removal processes are both accelerated, but overall more transfer takes place than removal because lubrication suppresses film removal. Previous studies which investigated dry testing of PEEK have also reported that tribological test conditions affected the dynamic balance of transfer and removal of PEEK

films on steel counterparts [19,22]. This study brings further clarification into the effects of lubrication. It shows the impact of lubrication not only on transfer but also removal of films, and its dependence on the operating conditions (SRR).

4. Conclusions

The mechanism of lubrication of the PEEK/steel contact with a PAO base oil has been investigated by focusing on two key factors: the formation of PEEK transfer films on the steel counterparts and the changes in hardness (softening) of PEEK specimens. The following conclusions have been drawn:

(1) Lubrication with PAO reduced the friction of the PEEK/steel contact regardless of the operating conditions. By contrast, the lubrication effects (positive or negative) on wear of PEEK depended on SRR;

(2) Lubrication with PAO had a softening effect on the wear track of PEEK, but no correlation was established with the wear rates of PEEK;

(3) The thickness of PEEK transfer films on the steel counter surface was the main parameter that controlled PEEK wear rates;

(4) Lubrication with PAO not only inhibited the transfer but also suppressed the removal of the PEEK films. This explains why lubrication can play both a positive or negative role on the wear of PEEK depending on the operating conditions.

This study elucidated the lubrication mechanism of PEEK and established the reason why lubrication showed positive or negative effects depending on the tribological conditions. This knowledge is fundamental for the design of efficient tribological systems and the formulation of suitable lubricants for PEEK/steel applications.

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Table Captions

Table 1   Tribological test conditions

Figure Captions

Fig. 1   MTM (a) schematic configuration and (b) set-up appearance

Fig. 2   Friction coefficient as a function of test time

Fig. 3   Friction coefficient averaged during the last ten minutes and wear rates of PEEK plates

Fig. 4   (a-h) Optical images and (i-l) nanoindentation hardness of after-test PEEK plates

Fig. 5   Nanoindentation hardness of after-test PEEK plates at 2 μm indentation depth

Fig. 6   (a-d) Optical images and (e-h) EPMA carbon maps of after-test steel balls

Fig. 7   XPS C 1s spectra of (a) unused PEEK plate and (b-e) after-test steel balls

Fig. 8   Friction coefficient as a function of test time

Fig. 9   Friction coefficient averaged during the last ten minutes and wear rates of PEEK plates

Fig.10   EPMA carbon maps of (a) preconditioned ball and (b, c) after-test steel balls with preconditioned steel balls
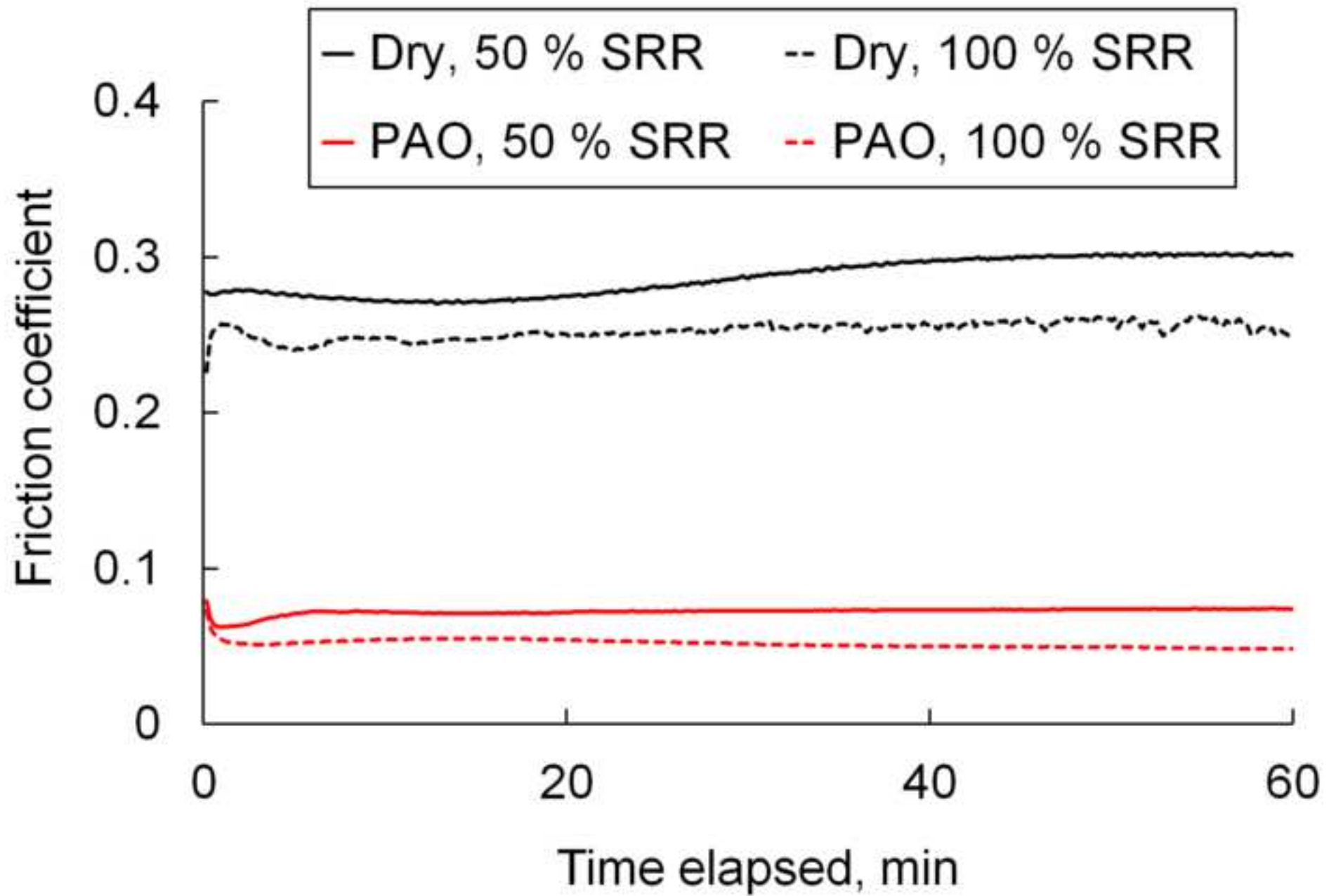
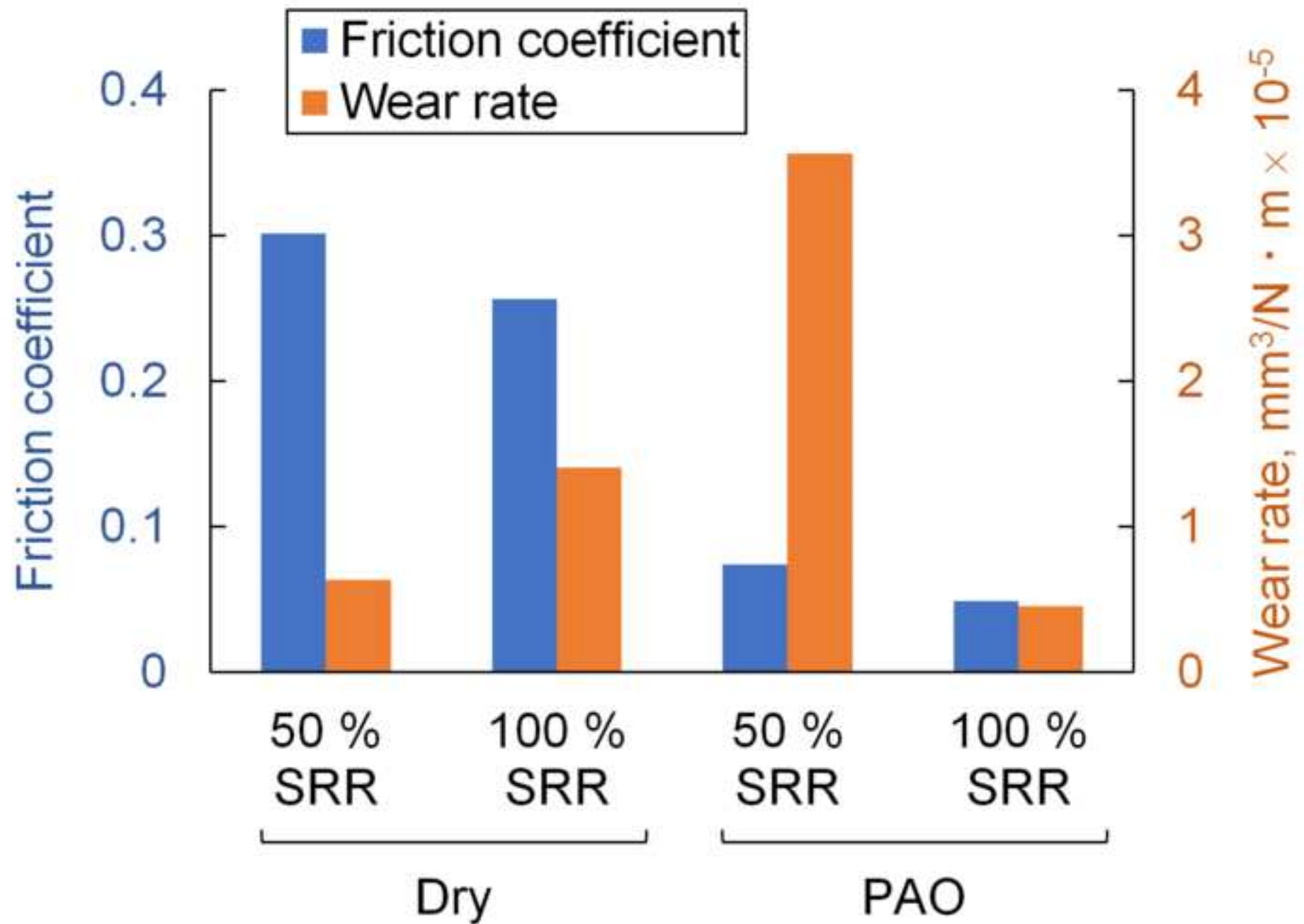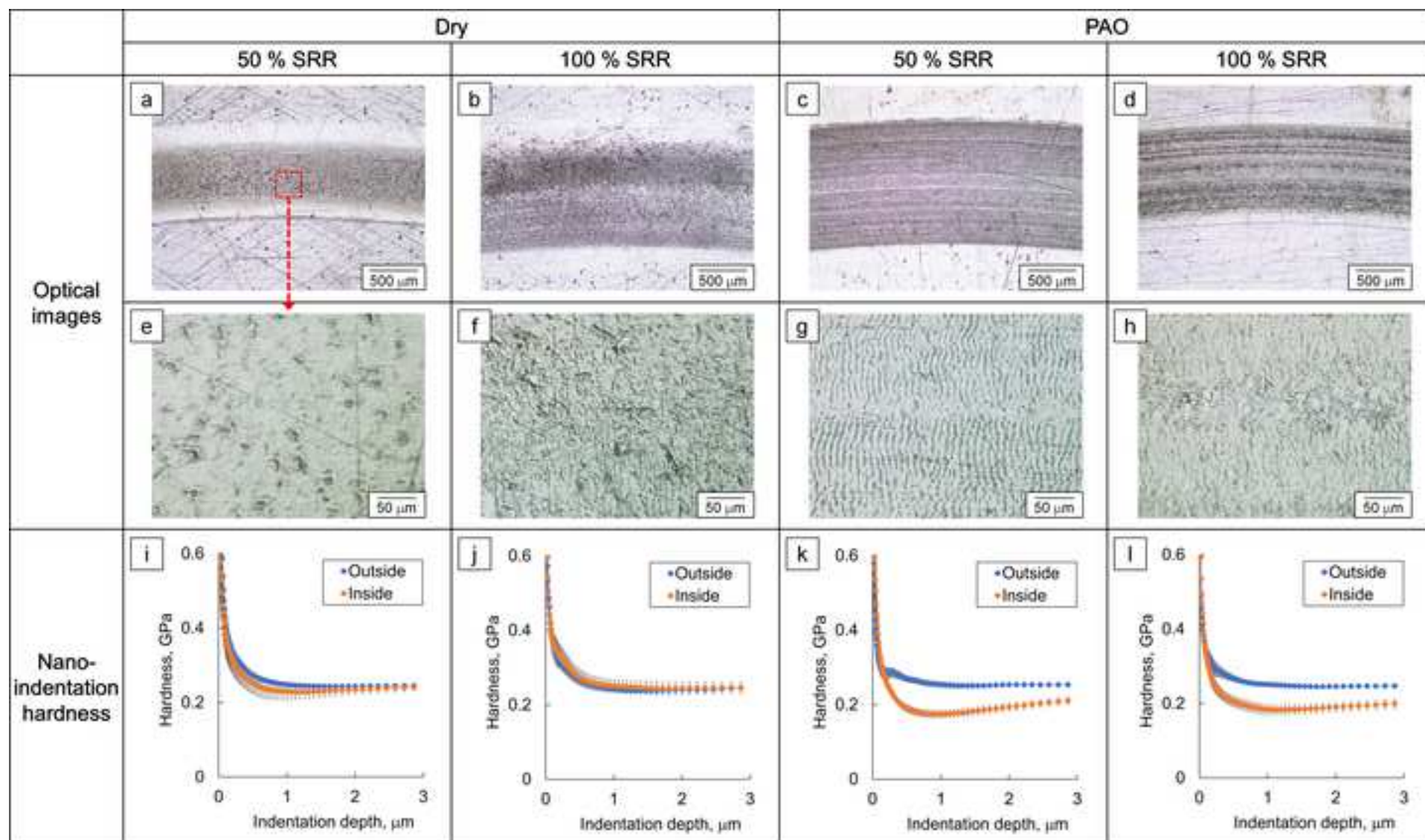Fig.11   Schematic of assumed mechanism of lubrication

Table 1

Test condition	Dry		PAO	
SRR	50 %	100 %	50 %	100 %
Entrainment speed	0.5 m s^{-1}			
Load / P_{max}	50 N / 0.16 GPa			
Temperature	Ambient (~25 °C)			
Testing time	60 minutes			
Lambda ratio	-		~0.2	

Figure 1   Click here to access/download;Figure;Fig 1.tif ↨

a)

PEEK   Steel

Dry or PAO

b)

Surface analyses

Wear and hardness measurements

Figure 2

Figure 2

Figure 3
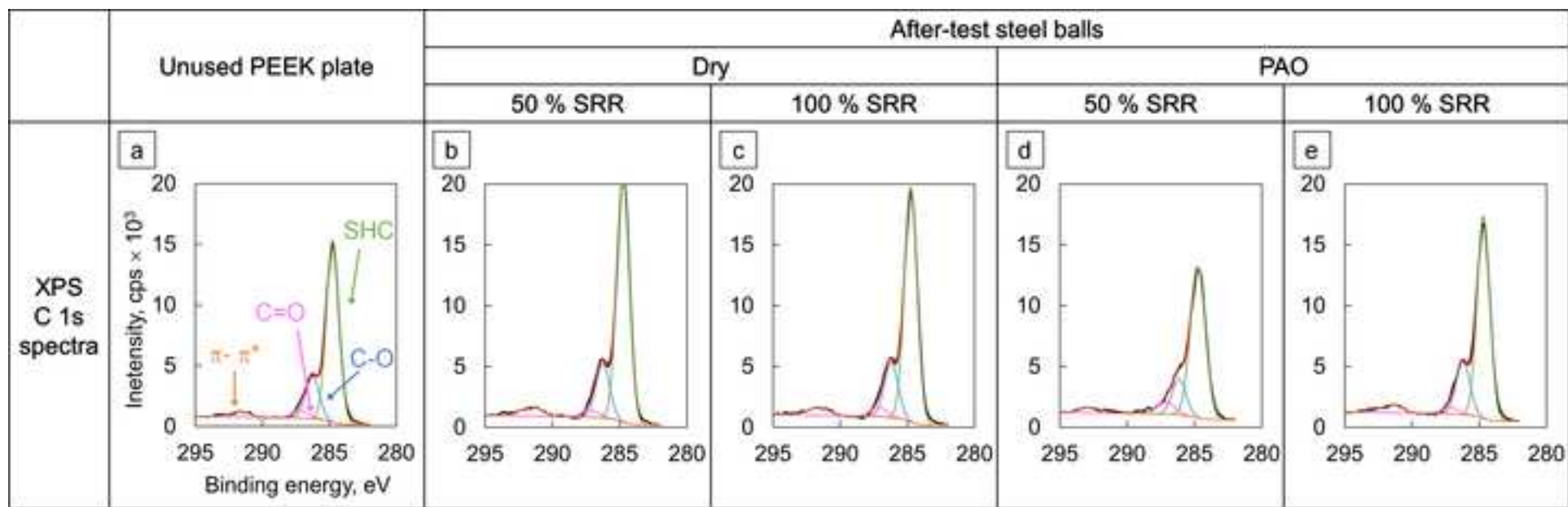
Figure 4

Figure 4

Figure 5

Figure 6
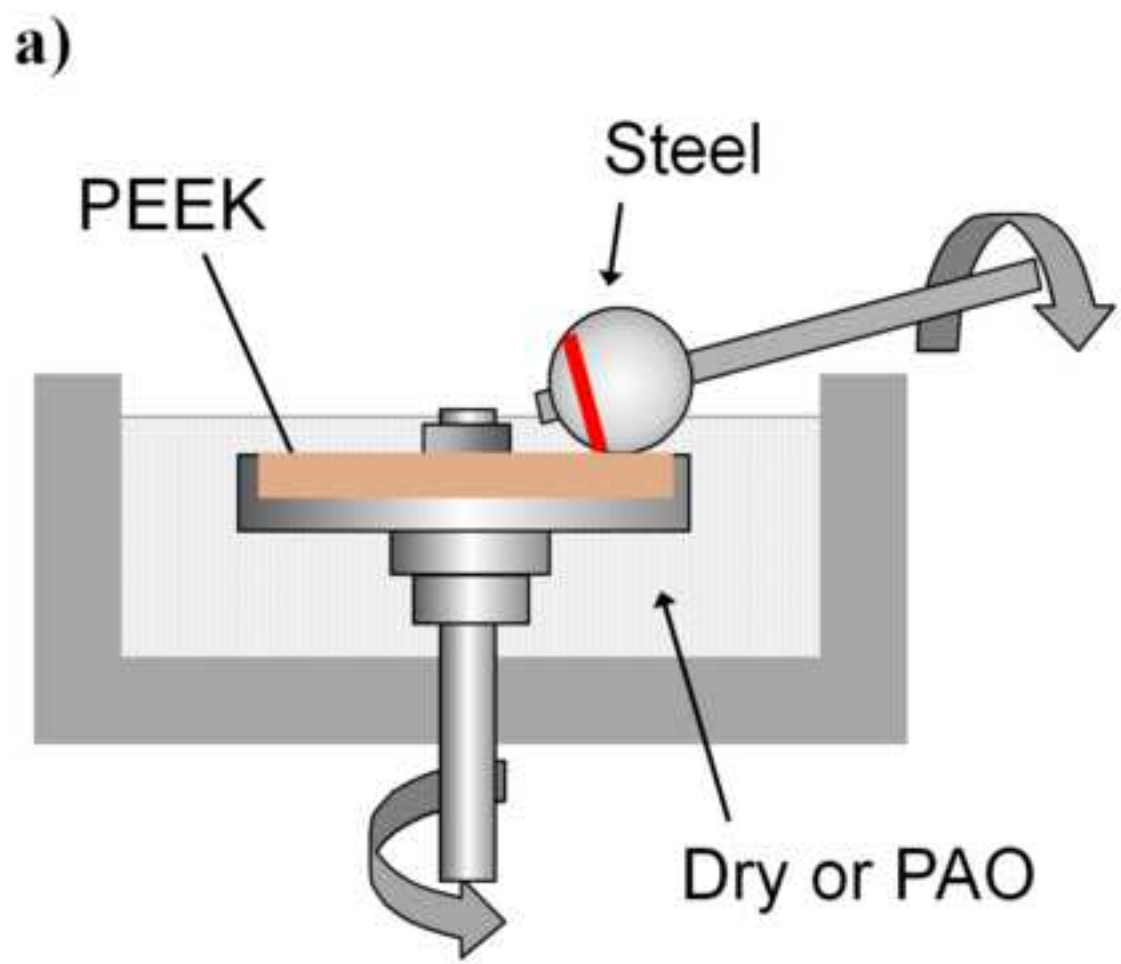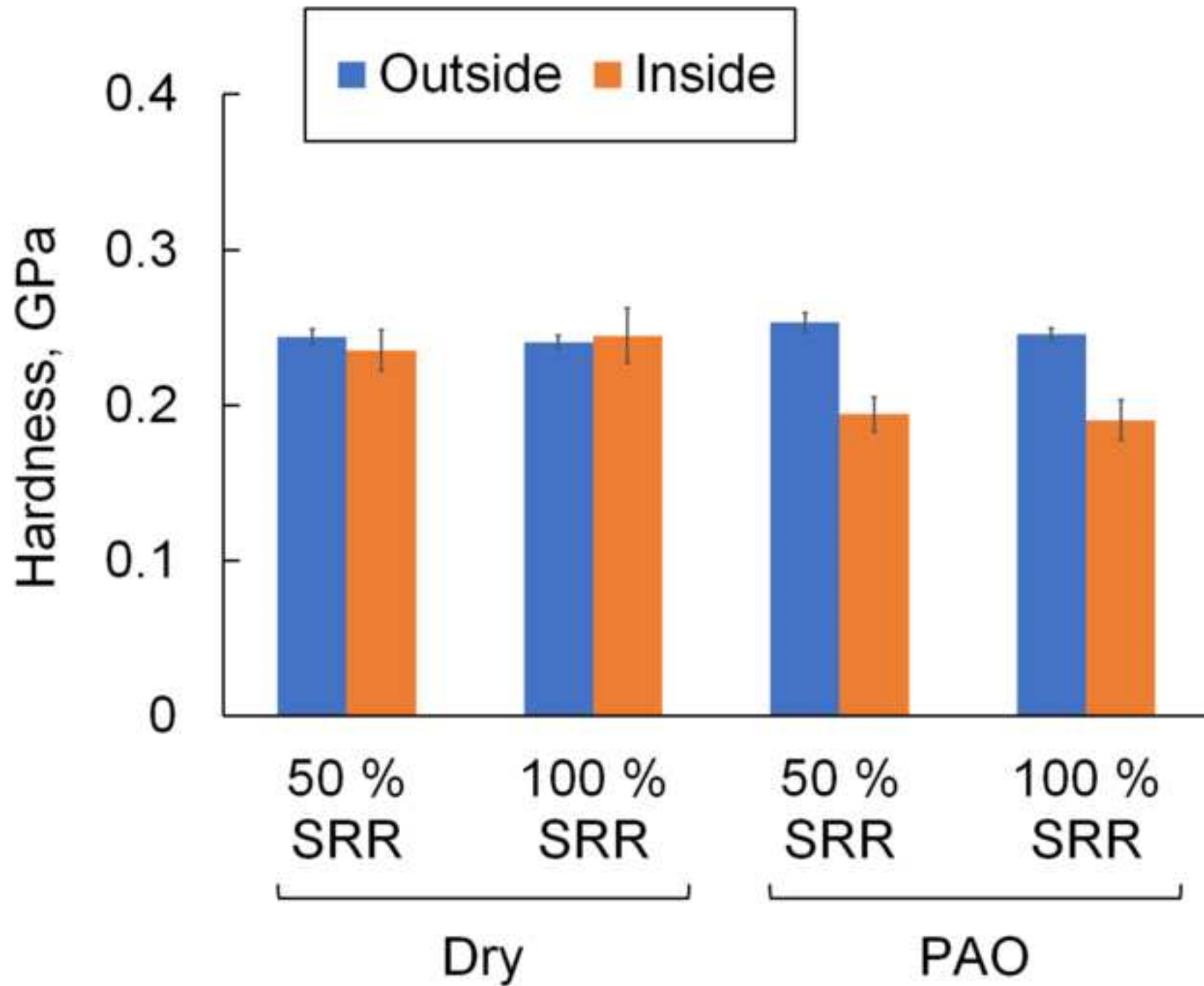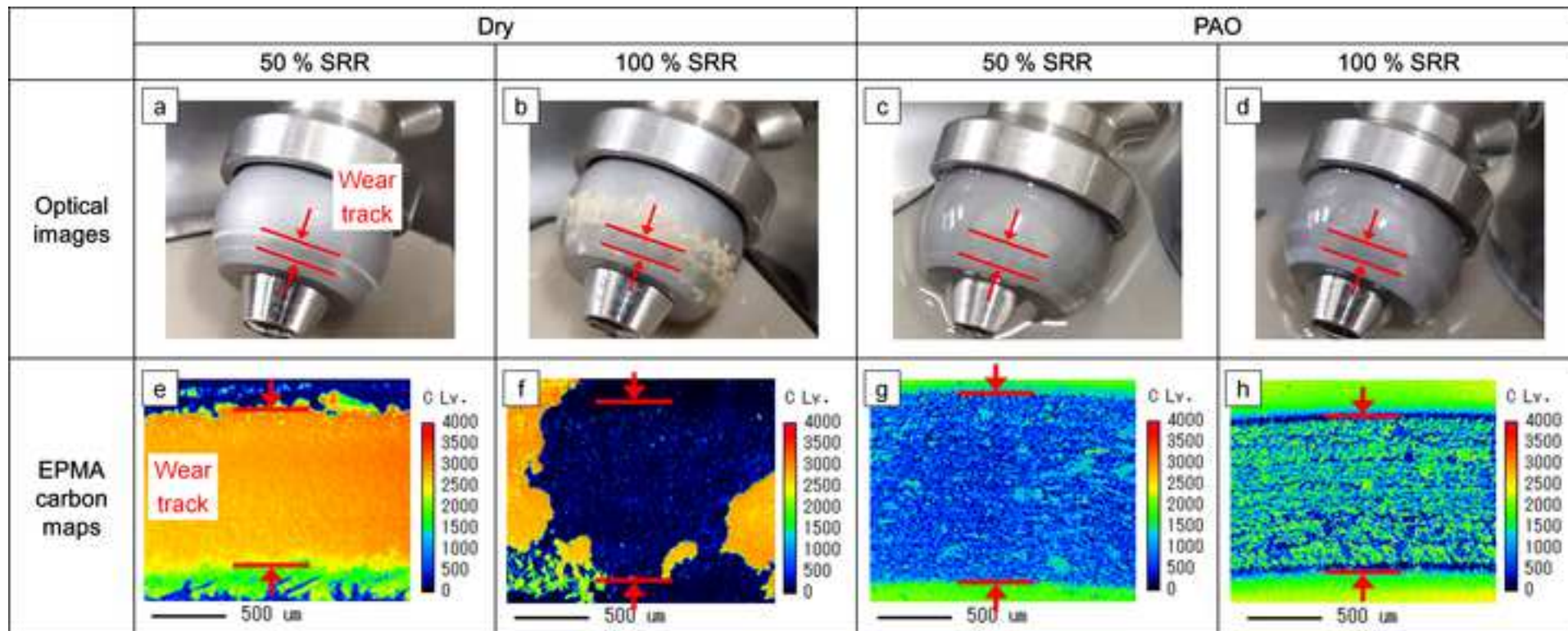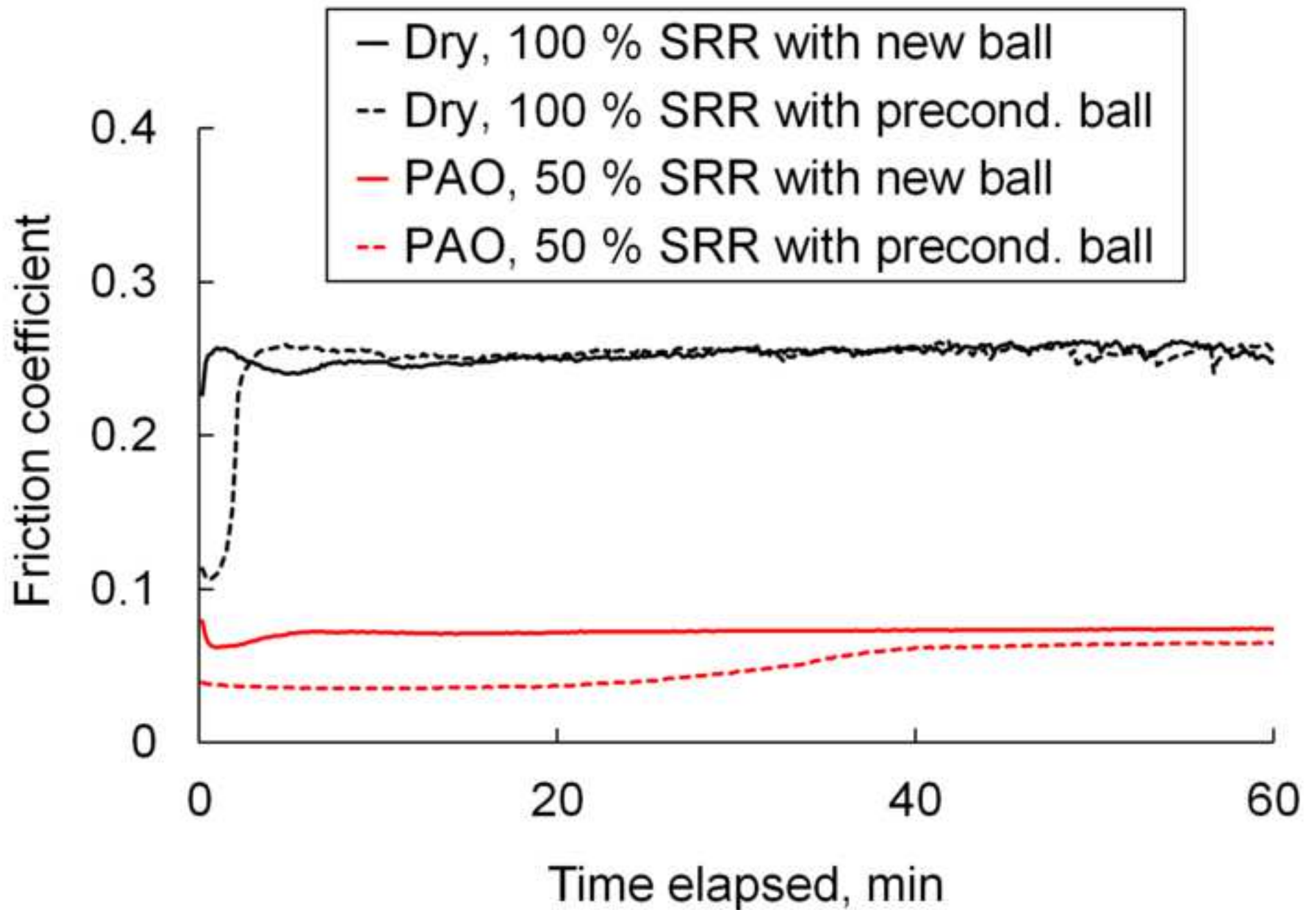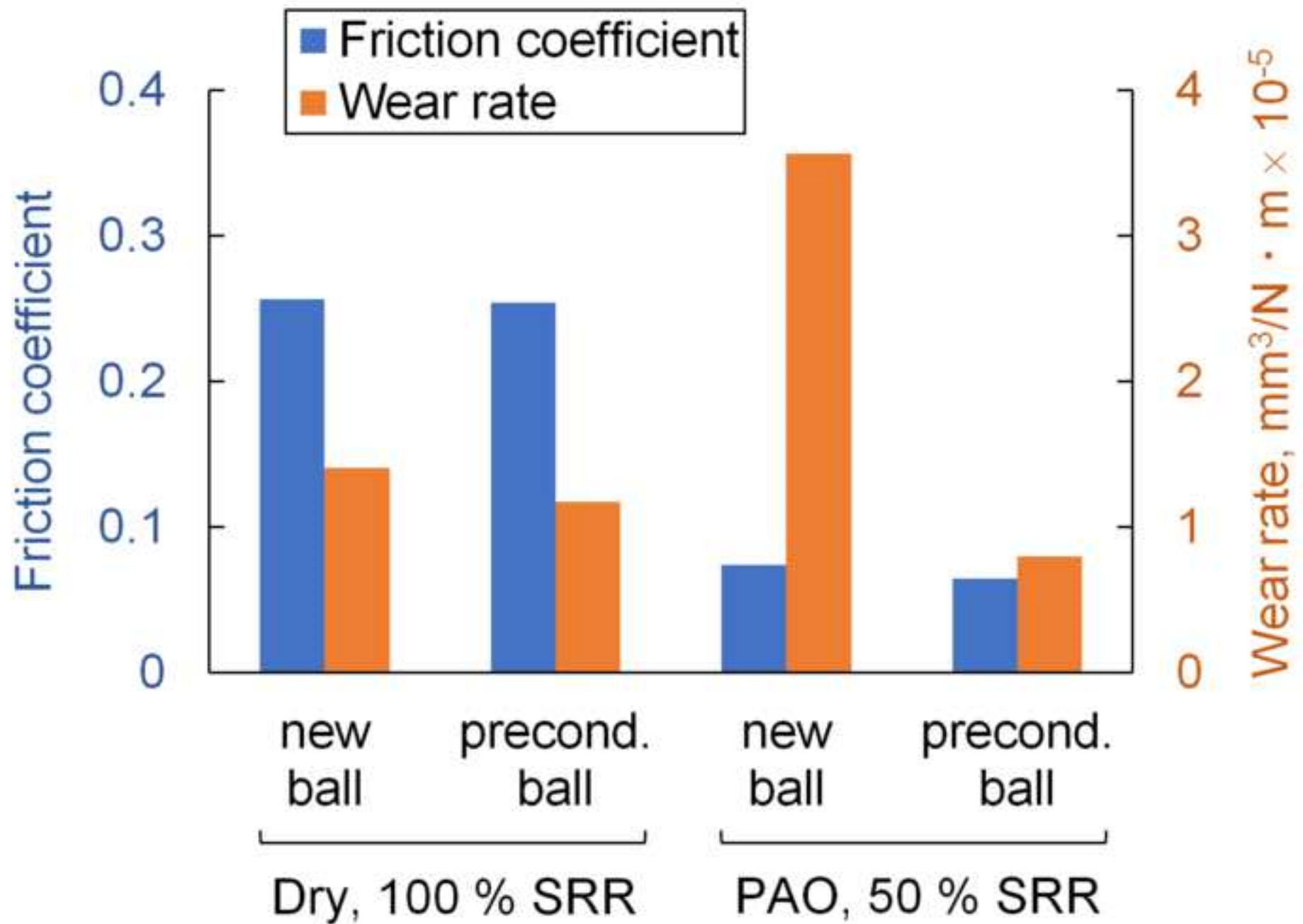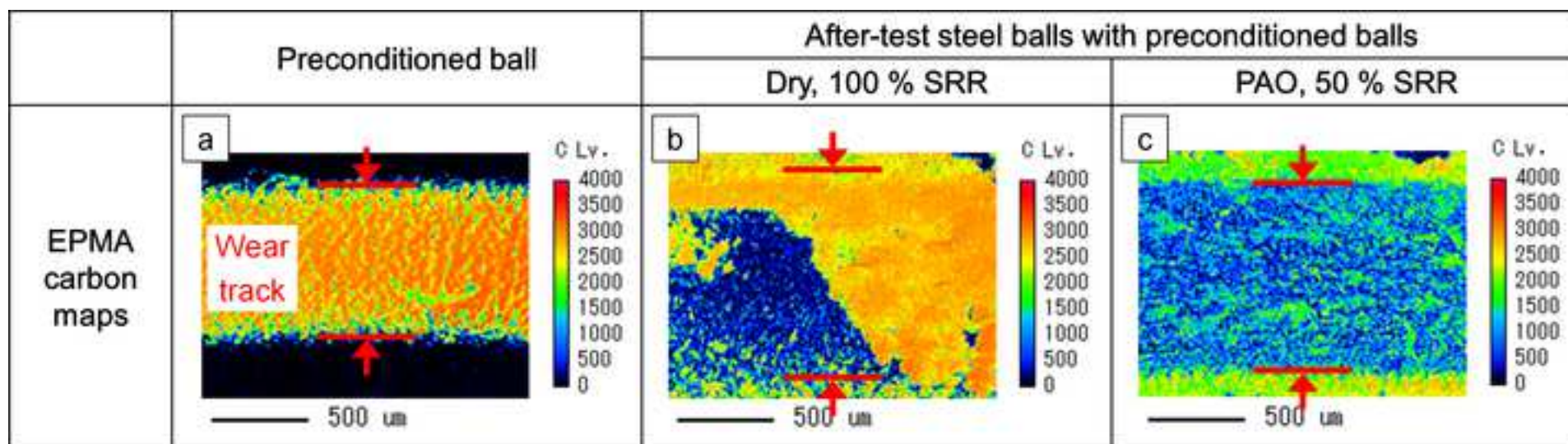
	Dry		PAO	
	50 % SRR	100 % SRR	50 % SRR	100 % SRR
Optical images	a	b	c	d
EPMA carbon maps	e	f	g	h

Figure 7   Click here to access/download;Figure;Fig 7.tiff ⬇

Figure 7

Figure 8

Figure 8

Figure 9　　　　Click here to access/download;Figure;Fig 9.tif ⬇

Figure 10

Click here to access/download;Figure;Fig 10.tif

	Preconditioned ball	After-test steel balls with preconditioned balls	
		Dry, 100 % SRR	PAO, 50 % SRR
EPMA carbon maps	a Wear track — 500 um	b — 500 um	c — 500 um

Figure 11